

NBSIR 74-456

Evaluation of the Fire Performance of a Dibromotetrafluoroethane - Blown Rigid Polyurethane Foam

T. G. Lee, W. J. Parker and M. Tryon

Fire Technology Division
Institute for Applied Technology
National Bureau of Standards
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Final Report

Principal Sponsor
Naval Ship Research and Development Center
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T. G. Lee, W. J. Parker and M. Tryon

The fire performance characteristics of a dibromotetrafluoroethane - blown rigid polyurethane foam were measured by several laboratory test methods. Measurements included: surface flammability, smoke and gases generated at elevated temperature and during combustion, ignition temperature, rate of heat release, and fire growth. The maximum concentration of the blowing agent in the specimen was approximately 13 Wt%. Specimen density was 0.046 g/cm^3 . As a function of temperature, release of blowing agent from collapsed cells began at about 60°C and became considerable at 135°C . The material had a flame spread index (ASTM E-162) of 11 with smoke levels of 170 and 480 (maximum specific optical density) under non-flaming and flaming exposures, respectively. The measured rate of heat release was 8.8 W/cm^2 , about 5 times that of a fibrous glass insulation. The measured flash ignition temperature was 530°C for the material.

Key words: Dibromotetrafluoroethane; flame spread index; fire tests: heat release rate; ignition temperature; rigid urethane foam; smoke.

1. INTRODUCTION

In the evaluation of the fire performance of structural rigid polyurethane foam material which may be directly exposed to potential shipboard fires, consideration should be given to the following characteristics:

- Surface Flammability
- Smoke and Toxic Products Generated During Combustion
- Release of Fluorocarbon Blowing Agent Relative to Temperature
- Ignition Temperature
- Ease of Ignition
- Rate of Heat Release
- Fire Growth Potential

While it is not possible to relate any of these characteristics directly and quantitatively to a life hazard under all use conditions, test methods now exist or can be adapted for comparative evaluation of these properties. Smoke generation, surface flammability, rate of heat release, and ignition temperature can be determined with relative ease using small-scale specimens and recognized standardized test methods.

Another potential hazard not directly related to fire is the possible decomposition of the foam at temperatures slightly above ambient and the release of blowing agent which is an integral part of the foam system.

Because the agent used in this foam is considered toxic* at some concentrations and since the fire properties of the foam are dependent in part on the blowing agent, information on its rate of release at elevated temperature was also obtained. This study was sponsored by the Naval Ship Research and Development Center, Carderock, Maryland.

2. MATERIALS

The closed cell rigid polyurethane foam was polymerized from resin and blowing agent by the sponsor into crude slab sections roughly 1.3 by 1.3 m with a thickness of 20 cm. The blowing agent, which is trapped in the closed cell structure of the foam, was a fluorocarbon compound, 1,2 - dibromotetrafluoroethane (fluorocarbon 114B2 or Halon 2402), an excellent fire retardant agent. Although the exterior surfaces varied considerably, the interior of the large specimen was fairly uniform in density with typical cell diameter of about 1 mm. The measured bulk density, based on an average of five samples, was 0.046 g/cm^3 (2.9 lb/ft^3).

A second specimen, labeled "aged" and fabricated on October 5, 1971 by the sponsor; appeared to have somewhat smaller cell diameter but a similar density of 0.046 g/cm^3 (2.9 lb/ft^3).

3. SPECIMEN PREPARATION

Specimens of uniform density were selected, cut into the appropriate sizes, and then heated in an oven at 60°C (140°F) at ambient pressure for 24 hours. This was followed by conditioning to equilibrium with an atmosphere maintained at 23°C (73°F) and 50% relative humidity. This heating and conditioning procedure was used with all the specimens for the surface flammability, smoke and ignition temperature tests. For other tests, specimens were tested as received or after conditioning by other methods to be described.

4. TEST METHODS AND PROCEDURES

4.1 Surface Flammability (ASTM E 162)

Flame spread was measured using the radiant panel apparatus (ASTM E 162)[1]**. In this test, a specimen 45.7 by 15 by 2.5 cm (18 by 6 by 1 in) thick was mounted in a holder and placed lengthwise at an angle of 30° with respect to the vertically mounted radiant panel. The distance between the specimen and the panel was least at the top of the specimen, about 12 cm ($4 \frac{3}{4}$ in). The energy output of the panel

*Freon Product Information B-2, E. I. du Pont de Nemours & Co., Wilmington, Delaware, 1969. Threshold Limit Value is 1000 ppm, LC 50 = 5%. More recent information show that concentration above 1000 ppm is considered unsafe for human exposure due to possible cardiac arrhythmias.

**Figures in brackets indicate the literature references at the end of this paper.

was controlled so that it was equal to the output of a blackbody of the same dimensions operating at 670°C.

An acetylene pilot flame was applied near the top of the specimen and the times for the flame front to travel in three inch increments (t_3 , t_6 , t_9 , t_{12} , t_{15}) were recorded. In all cases the test ended when the flame front traversed the whole length of the specimen, or after 15 minutes exposure time, whichever was shorter. The temperature rise was measured by thermocouples placed in the stack above the specimen.

The flame spread index, I_s , is calculated using the equation $I_s = F_s Q$, where Q is the heat evolved and F_s is the flame spread factor:

$F_s = 1 + 1/(t_6 - t_3) + 1/(t_9 - t_6) + 1/(t_{12} - t_9) + 1/(t_{15} - t_{12})$. The heat evolved is proportional to the observed maximum temperature rise of the stack thermocouples.

4.2 Smoke and Combustion Products

Tests were conducted using the Smoke Density Chamber as described in Appendix II, NBS Technical Note 708 [2]. The smoke produced during the burning of test specimens was measured photometrically, employing a laboratory test method developed for the purpose. The test utilizes a closed chamber of 0.5 m³ (18 ft³) volume containing an electrically heated furnace which provides an irradiance of 2.5 W/cm² on the surface of a nominal 7.6 cm (3 in) square specimen. The method assumes the applicability of Bouguer's law to the attenuation of light by smoke. Smoke quantity is therefore reported in terms of optical density rather than light absorbance. Optical density is the single measurement most characteristic of a "quantity of smoke" with regard to visual obscuration.

To take into account the optical path length, L , the volume, V , and the specimen surface area producing smoke, A , a specific optical density is defined as $D_s = V/LA (\log_{10} 100/T)$, where T is the percent light transmittance. Thus, for a selected exposure in the test chamber, and within certain limitations, a single test permits rough extrapolation to other surface areas and chamber volumes.

Specimens are subjected to two modes of exposure conditions, namely flaming and non-flaming. Both conditions can be considered typical in a real fire situation. Under the non-flaming exposure mode, the specimen receives an average irradiance of 2.5 W/cm² on its exposed surface to cause pyrolysis and emission of smoke. Under the flaming mode, in addition to the same surface irradiance, the specimen is subjected to pilot flames consisting of 6 small (9.5 mm length) premixed air-propane flamelets impinging on the base of the specimen.

Indications of the (maximum) concentrations of potentially toxic combustion products HBr, HCN, CO, HF, and NO_x were obtained by using commercial colorimetric detector tubes and the techniques described by Gross, et. al. [3]. This reference provides background on the use and limitations of the smoke and combustion products measurement methods.

4.3 Fluorocarbon Content of Foam Specimen

The weight concentration of fluorocarbon blowing agent, dibromotetrafluoroethane, in the foam system was measured for various specimens. These included the "aged" specimen, and new specimens subjected to selected preheating levels. An approximate rate of blowing agent loss from the specimen at 100°C was also determined.

The general procedure used for fluorocarbon analysis was gas chromatography of gaseous samples taken from a closed container in which the sample had been decomposed thermally. Initial evacuation of the container was necessary to prevent excessive pressure buildup during the pyrolysis of the foam. After sample pyrolysis in the container, a valved syringe was used to transfer a small volume of the vapor products to a gas chromatograph for analysis. The gas chromatograph was equipped with a thermal conductivity detector and a Porapak Q column held at 170°C. The response was calibrated by injecting known weights of the fluorocarbon and determining peak heights. A plot of peak height versus weight of sample served as a calibration curve.

The required samples were about 24 mm in diameter and 33 mm in length. Each sample was weighed and placed in the 100 ml volume quartz container. A mechanical pump connected to a syringe needle in the septum of the container, was used to evacuate the container for 30 minutes prior to heating. The sample was decomposed by heating with a burner flame until the foam collapsed and dense fumes filled the vessel. The container was cooled to room temperature and samples were analyzed in duplicate. Thus data were based on the average of two specimens, each using the above procedures.

The rate experiments were conducted in a similar manner except for the pyrolysis step. Larger samples were used and the containers held in an oven for various periods of time at a temperature of 100°C. Vapor samples were taken at the beginning and at various times. The rates were determined from the initial slopes of the time-concentration curves.

4.4 Ignition Temperature

The flash-ignition temperature of the foam was determined by using the test procedure of ASTM D 1929-68 [4]. Flash-ignition is defined as the lowest initial temperature of air passing around the specimen at which a sufficient amount of combustible gas is evolved to be ignited by a small external pilot flame.

The apparatus consists of a hot-air ignition furnace with a vertical ceramic furnace tube having an inside diameter of about 75 mm. A specimen weighing 3 ± 0.5 g is placed in a specimen pan located at the center of the furnace tube. The supply of air from the bottom of the tube passing the specimen is maintained at a velocity of either 1.5 m (5 ft)/min or 3 m (10 ft)/min and at the prevailing temperature of the furnace.

A small pilot flame located at the top of the furnace serves to ignite the combustible products from the specimen. After the specimen is in place, the temperature in the furnace tube is increased at a rate of about 60°C/min.

Temperature rise is continuously measured by a thermocouple placed at the center of the specimen. The temperature at which flaming ignition occurs, is reported as the flash ignition temperature.

4.5 Ease of Ignition

The ease of ignition test [5] utilizes a pair of 2.5 cm (1 in) thick specimens 14 by 15 cm (5-1/2 by 6 in) facing each other and separated by a distance of 2.2 cm (7/8 in). A flat-shaped premixed-flame, somewhat larger in size than the specimen and located in the space between the two specimens, provides flame contact corresponding to a heat flux of 3 W/cm². The time to sustained ignition, i.e. the exposure time required to produce sustained flaming, is found by a trial and error method. The specimen is exposed for a predetermined period of time and the presence or absence of flaming is noted. The presence of flame at any point on the specimen one minute after the exposure flame has been removed constitutes sustained ignition. The exposure time is then increased or decreased appropriately until the ignition time is bracketed.

4.6 Heat Release Rate

In the heat release rate calorimeter [6], the front surface of a 2.5 cm (1 in) thick vertical specimen 11.4 by 15 cm (4-1/2 by 6 in) is exposed to thermal radiation at a 3.3 or 6 W/cm² level from three gas-fired radiant panels situated at the front and both sides of the specimen. The edges of the specimen are shielded and the rear surface is separated from a water cooled brass block by an air space. The brass block constitutes a small section of the wall behind the one represented by the specimen. The heat removed from the rear surface of the specimen is measured by the rate of temperature rise in the cooling water. A propane burner near the flue of the calorimeter produces heat at a considerably greater rate than that of the burning specimen. When the specimen is burning, the propane flow is automatically reduced by the amount necessary to maintain the flue gas temperature constant. The rate of heat release through the front surface of the specimen is determined from the reduction in propane flow. Only heat released at the front surface is quoted in this report.

4.7 Fire Growth

Specimens of the test foam, 2.5 and 5 cm in thickness and 76 cm (30 in) square, were tested in the ceiling location of a rectangular enclosure. This enclosure has a 76 cm square base and height of 81 cm. It represents a small model of a compartment, in which the appropriate heat and mass flow rates from full-scale compartment fires have been scaled. This evaluation test is still under development and no published references are available at present.

Natural gas supplied to a diffusion-flame burner in the compartment was released through a 10 cm square horizontal porous plate located at the left corner of the compartment opposite the opening. The air for the burner as well as for the combustible ceiling was supplied by natural convection through the 18 cm wide and 69 cm high opening at the front of the compartment. The surface of the burner was positioned at 7.6 cm above the floor in the first test with a gas flow rate of 15.1 l/min, and 28 cm above the floor in the second test with a gas flow rate of 8.5 l/min.

The radiant flux near the floor, the air temperature just below the ceiling and the oxygen concentration of the air leaving the doorway were measured and compared with the same measurements for other ceiling materials.

5. TEST RESULTS

5.1 Surface Flammability

A total of 4 specimens was tested for flame spread. Two specimens were heated according to the standard procedures at 60°C prior to the test. The other two were heated at 135°C to examine the effect of partial loss of blowing agent.

The specimen heated at 135°C showed a color change, from light beige to dark brown, with a volume shrinkage on the order of 5-10%; the specimen heated at the standard 60°C showed very little apparent change.

Upon exposure to the radiant panel, all foam specimens immediately began shrinking and decomposing with rapid release of white smoke and gases which burned actively above the pilot burner with a yellowish flame. On two occasions the decomposition products were able to extinguish the pilot burner which was immediately relighted by the operator. In about 1.5 minutes, there were flashes of flame near the surface at the top of the specimen and after 2 minutes flashes extended momentarily down to 3, 6 and 9 inches. There was no sustained and continuous flaming. Observable flashes lasted less than 2 seconds, each with a frequency of about 5 to 10 per minute. Charring of the specimen progressed downward and reached a maximum at the 12 inch level in about 1.5 minutes.

The test flame spread factor is based on the measured times for sustained flaming. Since no sustained flames were observed, the factor is reported as 1 in accordance with the test method.

Because of the inhibiting nature of the fluorocarbon, flammability of the specimen was reduced considerably compared to rigid foam of conventional formulation.

The calculated flame spread index, $I_s = F_s \times Q$; flame spread factor, F_s ; and heat evolved, Q are shown in table 1.

Table 1. Flame Spread Index of Tested Foam Specimens

Specimen	Preheat °C	Fs	Q	Is
A	60	1	10.0	10
B	60	1	11.4	11
C	135	1	11.1	11
D	135	1	10.4	10

5.2 Smoke and Combustion Products

The measurements of smoke and gaseous products from specimens preheated at 60°C and 135°C are summarized in table 2. Similar to other types of rigid foam, smoke generation was higher under flaming than under non-flaming exposure in the Smoke Density Chamber. However, the smoke levels were higher than those for rigid foams based on other blowing agents.

Specimens preheated to 135°C for 24 hours showed lower smoke values compared to those preheated to 60°C. When heated to 135°C, weak cells in the specimen appeared to rupture and cause volume shrinkage and some color change. There was also a weight loss averaging about 30% as a result of the higher preheat temperature. Losses of some of the fluorocarbon blowing agent may account for the lower level of smoke (averaging 38%) and gaseous products.

The colorimetric tubes used to detect HBr are also sensitive to HCl. It was assumed that only negligible HCl was present because of the chemical composition of the foam and its blowing agent.

Table 2. Smoke and Combustion Products
from Specimens in Smoke Density Chamber

Combustion Products, ppm								
Preheat °C	Exposure	Initial Weight** g	Smoke Dm*	HBr	HCN	CO	NO + NO ₂	HF
60	Non-Flaming	6.5	173	-	-	-		-
		6.5	185	18	5	180		3
		6.2	148	25	-	-		-
		6.2	163	22	9	360		-
	Avg.		168	22	7	270		3
	60	Flaming	6.8	457	40	20	700	-
6.7			478	30	27	850	22	-
6.4			470	40	25	-	-	30
6.4			513	50	55	900	15	-
Avg.			480	40	32	820	18	30
135	Non-Flaming	4.2	158	12	6	260	-	-
		4.7	158	4	5	-	1	-
	Avg.		158	8	6	260	1	
135	Flaming	4.7	346	12	18	600	-	-
		4.5	273	10	20	600	30	-
		4.3	268	10	16	500	30	-
	Avg.		268	11	18	570	30	

*Maximum Specific Optical Density (corrected)

**All specimens were 2.5 cm (1 in.) thick prior to preheating.

5.3 Loss of Dibromotetrafluoroethane Blowing Agent

The weight percentage of dibromotetrafluoroethane in the foam specimens was determined by the method outlined in paragraph 4.3. Weight loss as a result of preheating conditions was based on the difference in weight relative to the original weight. Specimens were sufficiently large in size to minimize errors.

Table 3 summarizes the results of weight loss due to preheating and the decrease concentration of $C_2F_4Br_2$ in the conditioned specimens. The data were based on averages of at least 2 measurements. The maximum $C_2F_4Br_2$ in the specimen was on the order of 13% by weight.

Table 3. Weight-Loss and Percent (Wt.)
of $C_2F_4Br_2$ in Specimen After Preheating

Specimen	Preheating Temp °C	Duration Hrs.	Specimen Weight Loss %	Wt. Concentration $C_2F_4Br_2$ %
Recent	None		0	13.0
Aged (Oct. 71)	None		0	11.9
Recent	60	24.0	1.2	*
Recent	135	24.0	11.8	7.6
Recent	200	0.5	27.0	-
Recent	200	2.5	35.0	0
In Service "Box 1"	None		0	13.0
In Service "Box 2"	None		0	11.7
In Service "Box 3"	None		0	13.2

* Not measured, estimated approximately 12%.

The rate of blowing agent release at a temperature of 100°C was also determined. It was based on the initial slope of the $C_2F_4Br_2$ concentration-time plot taken at about 10 minute intervals for a period of 40 minutes. The rate of release of blowing agent was on the order of 3 Wt% per hour. This is a fairly crude estimate although based on several experimental measurements.

It is apparent that at elevated temperature, there is a gradual loss of the blowing agent. At 60°C for 24 hours, a total weight loss of about 1 Wt% was detected. At 100°C the rate of agent loss was estimated to be 3 Wt% per hour. At 135°C and 200°C there was a high rate of loss from the specimen. After 2.5 hours at 200°C, no agent was left in the specimen. However, specimens which had been in service for several years were found to have retained the initial 12 to 13% of blowing agent.

5.4 Flash-Ignition Temperature

Results of flash-ignition temperatures test of the foam specimen using the procedures of ASTM D1929, are shown in table 4.

Table 4. Flash-Ignition Temperature of Foam Specimen

Air Velocity ft/min	Flash-Ignition Temperature °C
5	550
10	510, 530

The above represent low and medium velocity flash-ignition temperatures.

Self-ignition temperature, is always higher than the flash-ignition temperature, though not measured in the test.

5.5 Ease of Ignition

Under the ease of ignition test method described in section 4.5, the specimen did not undergo sustained ignition. In three separate tests the specimens did not burn for a period of 1 minute or more after the exposure flame had been removed.

5.6 Heat Release Rate

Specimens of the foam (2.5 cm thick) were also tested in the NBS Heat Release Rate Calorimeter at irradiance levels of 3.3 and 6.0 W/cm². The results are tabulated in table 5. There was no flaming of the specimens at 3.3 W/cm² without a pilot. Flaming was observed for all other specimens tested.

Table 5. Heat Release Rate Calorimeter Measurements of the Test Foam

Irradiance W/cm ²	Pilot On/Off	Weight Loss %	Peak Heat Release Rate W/cm ²	Highest 1 min. Average Release Rate W/cm ²	Total Heat Released J/cm ²	Flaming Yes/No
6.0	Off	88.4	11.7	9.3	960	Yes
6.0	Off	89.4	12.2	9.2	890	Yes
6.0	Off	83.4	9.9	7.9	690	Yes
Average		87.0	11.2	8.8	850	
3.3	Off	77.9	4.1	4.1	480	No
3.3	Off	74.7	5.7	5.2	690	No
3.3	Off	74.2	5.0	4.3	620	No
Average		76.0	5.0	4.6	600	
3.3	On	77.2	4.7	4.6	620	Yes
3.3	On	77.8	8.6	6.4	-	Yes
3.3	On	78.1	7.9	7.7	550	Yes
3.3	On	78.4	7.2	5.7	620	Yes
Average		78.0	7.1	6.0	600	

For comparison, the average values of the heat release at 6.0 W/cm² irradiance for red oak, the test foam and fibrous glass insulation are shown in table 6.

Table 6. Heat Release Rate of the Test Foam and Other Materials at 6 W/cm²

Material	Speciman Thickness cm	Peak Heat Release Rate W/cm ²	Highest One Minute Average W/cm ²	Total Heat Release J/cm ²
Red Oak	2.0	18.0	18.0	11250
Test Foam	2.5	11.2	8.8	850
Fibrous Glass	2.5	2.1	1.7	350

The highest one minute average heat release rates at 6.0 W/cm² for the foam are about one half that of red oak, while the total heat released is an order of magnitude lower. However, the heat release rate of the foam is about 5 times that of fiber glass.

5.7 Fire Growth Experiments

The radiant flux at 7.6 cm above the floor, the air temperature 2.5 cm below the ceiling, and the oxygen concentration of the air leaving the top of the doorway for the test foam are compared to results for other ceiling

materials in table 7. The insulation board and the acoustic tile are both wood fiber insulation boards. All of the values reported in the table were the extreme values observed during the first 15 minutes of the test.

Comparison of test materials is valid only if the test exposure conditions in the model compartment are identical, i.e. test condition 1 or 2.

Under both exposure conditions the foam specimen contributed less to fire buildup in a room than did the fiber glass. However, the much larger value of heat release rate for the foam indicates that its contribution would become relatively greater as the exposure conditions become more severe. Furthermore, there was a vigorous generation of smoke from the test foam when it was first exposed to the flame from the elevated burner during the second set of test conditions.

Table 7. Comparison of the Test Foam with Other Ceiling Materials in the Model Compartment

Condition* Test	Material	Thickness cm	Radiant Heat Flux Near Floor W/cm ²	Air Temperature °C	Oxygen Level % of original
1	Test Foam	2.5	0.31	240	-
1	Fibrous Glass	2.5	0.40	450	-
1	Cellulose Insulation Board	2.5	0.80	580	-
2	Test Foam	2.5	0.19	330	78
2	Fibrous Glass	2.5	0.25	380	79
2	Acoustic Tile	2.5	0.81	750	16

* Condition 1, burner 7.6 cm from floor and gas flow rate of 15.1 l/min.
Condition 2, burner 28 cm from floor and gas flow rate of 8.5 l/min.

6. SUMMARY

Samples of a rigid urethane foam incorporating dibrometrafluoroethane as a blowing agent were subjected to seven laboratory measurements to evaluate its fire performance characteristics. Based on the test foam specimens submitted, the following results were obtained:

1. The weight concentration of the blowing agent in the foam was about 13%. Foam specimens that were aged in service appeared to retain the original concentration of fluoro-carbon agent.

2. Release of fluorocarbon blowing agent appears to initiate at a temperature as low as 60°C. The release rate was on the order of 3%/hr at 100°C. At temperatures of 135°C and above, a high rate of agent loss was noted.
3. Surface flammability in terms of a sustained flame spread index as defined by ASTM E-162, was about 11, low relative to other rigid foams. However, the foam showed tendencies to produce momentary flashing.
4. The maximum specific optical density of smoke generated was 170 and 480 for non-flaming and flaming exposure conditions, respectively. This is slightly higher than other rigid urethane foams of comparable density.
5. The flash-ignition temperature measured by the ASTM D 1929 test method was on the order of 530°C.
6. The highest one-minute average heat release rate measured by the heat release rate calorimeter at an irradiance level of 6.0 W/cm², was 8.8 W/cm² for the test foam. This compares with 18 W/cm² for the red oak and 1.7 W/cm² for fibrous glass insulation.
7. A compartment model experiment showed that the test foam contributed less to fire build-up than did fibrous glass insulation similarly located at the ceiling position. However, its vigorous generation of smoke and significant heat release rate may outweigh this advantage.

7. ACKNOWLEDGEMENTS

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16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.) The fire performance characteristics of a dibromotetrafluoroethane - blown rigid polyurethane foam were measured by several laboratory test methods. Measurements included: surface flammability, smoke and gases generated at elevated temperature and during combustion, ignition temperature, rate of heat release, and fire growth. The maximum concentration of the blowing agent in the specimen was approximately 13 Wt%. Specimen density was 0.046 g/cm ³ . As a function of temperature, release of blowing agent from collapsed cells began at about 60°C and became considerable at 135°C. The material had a flame spread index (ASTM E-162) of 11 with smoke levels of 170 and 480 (maximum specific optical density) under non-flaming and flaming exposures, respectively. The measured rate of heat release was 8.8 W/cm ² , about 5 times that of a fibrous glass insulation. The measured flash ignition temperature was 530°C for the material.			
17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons) Dibromotetrafluoroethane; flame spread index; fire tests; heat release rate; ignition temperature; rigid urethane foam; smoke.			
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